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TAPCO Div.
~~a division of~~
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Cleveland, Ohio

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MONTHLY PROGRESS REPORT

No. 4

for

October 1962

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Submitted by

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NEW PRODUCT RESEARCH of TAPCO

(NASA Contract NAS3-2551)

Schwartz

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OTS PRICE

XEROX $ 1.60 ph

MICROFILM $ .80 mf

Monthly Progress Report No. 4
November 14, 1962

I. INTRODUCTION

Indent in all cases & brackets

This document represents a fourth monthly report covering the work on the experimental program for development of an "Osmotic Still" and improvement in the performance characteristics of ~~the Ionics Dual~~ Membrane Fuel Cell during the month of October, 1962. This development work is being accomplished under NASA-Lewis Research Center Contract No. NAS 3-2551 by the New Product Research Department of Tapco and Ionics, Inc. as a subcontractor to Tapco.

II. OVERALL PROGRESS

A. Tapco Portion of Program

1. A total of four tests with Ionics membrane were performed during this month. Each of these tests ended in membrane failure by cracking prior to the establishment of conditions required for membrane performance tests.

2. With each succeeding test, modifications to the test unit were made and additional precautionary measures were taken to prevent membrane failure. This included, among other items, a redesign of perforated membrane supports (see Figure 2 of Progress Report No. 1 of August 16, 1962); use of Monel Metal Screen instead of Trilox material between the membrane and the perforated membrane supports; decreasing the amount of tension on the bolts around the periphery of the test unit to prevent the cracking of membrane within the gasket bearing areas. More details are given under Part V.

3. Short duration qualitative test was also performed to determine if 15 psi pressure differential across the membrane would force the liquid through a cation Ionics membrane without cracking it. Details of this test are described under Part V.

B. Ionics Portion of Program

1. Two single fuel cell units have been put on life test and have been performing for over 200 hours. These cells will be used as controls for further testing. Two other cells with "sintered" electrodes are scheduled to go on test the week of November 12, 1962.

2. Duplicate samples of platinized titanium, uncoated titanium, Tri-lok mesh, epoxy, buna N, Teflon and 9 oz. glass-backed membranes had been immersed in 6 N H_2SO_4 in three separate baths at 30^o, 60^o and 95^oC. The tests were discontinued after 550 hours and the materials weighed and examined. See Table I.

3. A literature survey was made of various plastics and metals for use in fuel cells either as a gasketing or as construction material for compartments, separator plates or pusher plates.

   Techniques for coating metals and plastics with noble metals in order to protect them from hot sulfuric acid were also investigated.

4. Test rig set-up: Control equipment for four ambient temperature (30°C) cells has been set up. This is capable of controlling temperature, pressure and flows of gases and electrolyte. The regulatory and control system for a high temperature device has been assembled.

5. Materials for making sintered electrodes were ordered and large sintered electrodes produced.

III. CURRENT PROBLEMS

A. Tapco Portion of Program

1. The most pressing current problem of this part of program to date is the difficulty in preventing the Ionics membranes from cracking under the imposed pressure differential across the membrane required by the test conditions. The steps taken to prevent membrane cracking are described in Part V.

B. Ionics Portion of Program

1. The prevention of attack by hot sulfuric acid on titanium.

2. The plugging of gas outlet lines of the cells with sulfuric acid and water.

3. Deterioration of cell performance due to lag in diffusion of H_2SO_4 into region of water formation at oxygen electrode.

4. Obtaining adequate contact between electrode and membrane in new fuel cell configuration.

IV. NEXT MONTH'S EFFORT

A. Tapco Portion of Program

1. Tests utilizing cation and anion membranes produced by manufacturers other than Ionics, Inc. will be performed. Preliminary studies indicate that these membranes are considerably stronger than those produced by Ionics, Inc. thereby minimizing membrane cracking problem.

B. Ionics Portion of Program

1. Test cells will be placed in operation at 30°C and 95°C. Variations in these fuel cells will include an attempt to reduce membrane thickness, and alteration of waterproofing content of sintered electrodes.

2. Use of gold plated titanium instead of platinized material in order to avoid attack of 6 $\underline{N}$ H_2SO_4 on cell separator and on pusher plates at elevated temperatures as indicated in the materials testing data in Table I.

3. Further materials testing at 30, 60, and 95°C.

4. Preliminary design efforts in reference to a 5-cell battery.

5. Due to the expected superiority of "sintered" electrode, control experiments at 30 and 95°C will be performed with these electrodes. As part of the control experiments, an attempt will be made to eliminate the corrosion problems arising from titanium degradation by utilizing electroplated gold film on separators and pressure plates.

V. TEST RESULTS

A. Tapco Portion of Program

1. The initial test was with cation membranes and with water used in place of the H_2SO_4 solution. As a vacuum was slowly drawn on the vapor side of the test unit, periodic leakage of water into the condenser indicated membrane failure at the onset of the test. Water temperature was raised to 180°F before taking membrane out of the test unit in order to expose the membrane to both temperature and pressure of about 15 psi.

   Inspection of the membranes after dismantling of the test unit revealed excessive cracking along the gasket edge of the membrane and considerable sagging of the membrane towards the vapor cavity as well as cracking under the gaskets.

2. Modifications to test unit prior to next test included making closer fitting support plates to give better support at gasket edges and also trimmed gasket I.D. to eliminate interference with spacer material.

   Cation membranes after soaking in 30% H_2SO_4 for approximately 118 hours was tested in the test unit for approximately 4-1/2 hours with H_2SO_4 temperature held at 175°F and the vacuum on the vapor side held at 48 cm Hg. No liquid entered condenser prior to test conditions producing condensation indicating no large leakage was present in the membrane. However, water collected had a pH of 1 indicating leaks. Inspection of membrane showed membrane sagging towards the vapor cavity with small cracks in the center. Membrane also had cracks under gaskets with bolts tightened to a torque of 50 in pounds.

3. Modification to test unit included increasing spacer dimensions from 0.56" to 0.59" and tightening of bolts to 35 in pounds.

   Cation membranes were tested but membrane failure was evident early in the heating up period as the condenser was filled with liquid within 50 minutes of starting time. Liquid in condenser was very acedic. Membrane inspection revealed many cracks in the center of membrane even though sagging was quite small.

4. Anion membranes were installed in the test unit and tested under same test conditions as above. Again, test was unsuccessful due to the same membrane failure as in the previous tests.

5. Modifications in test unit included replacement of Trilox spacer material with Monel screen (12 mesh .032 in. D. wire). Cation membranes were installed with bolts tightened to 25 in pounds.

   Testing membranes with H_2SO_4 temperatures of only 78°F resulted in acid leakage into condenser with up to 72.6 in. Hg vacuum on vapor side of membranes.

Inspection of membranes indicated no sagging at all in membrane surface but cracks were visible.

6. Previous tests were run with the membranes either soaked in 30% H_2SO_4 prior to installation or installed with only water soaking. Two more membrane tests were conducted but membranes were first progressively soaked in 10%, 20%, and 30% H_2SO_4 for approximately 30 minutes in each solution to eliminate possibility of cracks due to rapid shrinking of the membrane upon exposure to more concentrated solutions. Both membrane tests produced water with a pH of 1 and upon removal of membrane from unit inspection showed small cracks in center of membrane.

7. A qualitative test was performed to determine whether liquid could be forced through an uncracked Ionics membrane with a 15 psi pressure differential. A cation membrane was placed and secured over a 3/8 in. hole with water at 15 psig on one side, and atmospheric air on the other side. The air side of the membrane was covered to prevent H_2O evaporation of water that may leak through. The test was conducted over a period of 1 week and no H_2O was forced through the membrane at these operating conditions.

B. <u>Ionics Portion of Program</u>

1. Control and regulatory systems are being installed.

2. Life Tests: As mentioned in Section II, two cells of the new configuration, both containing paste type electrodes were placed "on stream". These cells have been operated continuously except for very short interruptions at a current of four amperes or a current density of 16 amperes per square foot. The data reported in Tables II and III indicate that operation is not uniform, as yet, possibly due to permeated liquid in gas compartments or variations in gas feeding rates or acid normality*. The voltage of cell 9718 has been significantly higher (100-150 millivolts) than that of cell 9719. This is probably a result of the use of more Tri-lok in cell 9718, thereby producing better catalyst-membrane contact. The most recent data, however, indicates that the difference between the two cells is no longer significant. Our best estimate of the situation is that the Tri-lok loses compressive ability after immersion in 6 <u>N</u> H_2SO_4 and contact between the catalyst and membrane relaxes.

3. The results of material testing are summarized in Table I. It is to be noted that platinized and plain Ti is not suitable for use at higher temperatures. Tri-lok epoxy and buna N can be used up to 60°C. Tests at elevated temperature have to be rerun to ascertain their usability under such conditions. The first set of 95°C tests could not be completed since oil from the bath leaked inside the containers.

* See note under Table III.

VI. QUALITY ASSURANCE

A. Tapco Portion of Program

As a result of a few instances of undetermined leakage paths within the Osmotic Still while on test, it is necessary to eliminate the possibility of the cause being in the membranes as received from the vendor, versus leakage through gasket and bolt hole areas in the unit.

A means for inspecting membranes for flaws, tears, holes, etc., at Receiving Inspection is under investigation. Dye penetrant, optical examination, polarized light, and pressure testing are some of the methods under consideration.

This type of inspection poses a problem, because of the handling techniques that will be required to keep the membranes moist at all times.

The most efficient and practical means at this time would seem to be a simple fixture to hold the membrane while applying a light pressure on one side using an appropriate gas as a medium. Bubble indications in the fluid on the reverse side would pin-point any flaws or voids in the material.

In the meantime a microscope at 10X will be used to examine all membrane material received until a more positive means is decided upon.

B. Ionics Portion of Program

The institution of Quality Assurance requirements of the contract continue.

Quality Control investigation of materials is also continuing. Spectrographic analysis of each platinum black lot is being received and a file maintained with reference to maximum particle size and other pertinent characteristics. Titanium, epoxy glass fiber plastic, and supporting plastics are now being segregated by lot number with final vendor test results obtained for each shipment where possible. Records of the preceding are being kept.

Finally, calibration of standards and maintenance of calibrated instruments is continuing.

TABLE I

Materials Testing at 30° and 60°C

Name of Material	Temp = 30°C Test Time = 550 hrs.			Temp = 60°C Test Time = 550 hrs.		
	% loss or gain in wt.	Color Change	Deformation	% loss or gain in wt.	Color Change	Deformation
Platinized titanium	- 0.4	none	none	- 11.0	gave blue solution	Pt coating came loose
Plain titanium	-40.0	gave blue solution	surface was generally attacked	-100.0	gave blue solution	specimen generally attacked
Epoxy	0.0	turned whiter	none	+ 8.4	none	none
Membrane (8 oz. glass-backed cation)	- 3.0	none	none	- 12.7	turned red-dish brown	none
Buna N	-12.1	very slight	slightly warped	- 5.5	gave a slightly yellowish solution	warped
Tri-lok	- 0.2	none	none	- 0.3	none	none
Teflon	- 0.8	none	none	- 0.8	none	none

TABLE II

Cell 9718 - Paste Electrode

Current:	16 amp/ft^2	Area:	0.25 ft^2
Electrolyte Pressure:	20 psig	Gas Pressures:	15 psig
Bath Temperature:	30°C		

Hours of Operation	Voltage
0	0.46 volt
0.5	0.65
11.3	0.78
20.8	0.78
41.2	0.72
71.5	0.72
118.0	0.68
143.5	0.64
165.8	0.60
186.0	0.59

TABLE III

Cell 9719 - Paste Electrode

Current	16 amp/ft^2	Area:	0.25 ft^2
Electrolyte Pressure:	20 psig	Gas Pressure:	15 psig
Bath Temperature:	30°C		

Hours of Operation	Voltage
0	0.58 volts
1.1	0.63
14.5	0.59
27.0	0.62
44.5	0.59
74.8	0.59
121.2	0.67*
146.2	0.46
169.0	0.50
188.8	0.63

* Normality of acid restored to 6N by additions to external electrolyte reservoir as required just before reading.

TAPCO a division of Thompson Ramo Wooldridge Inc.

FINANCIAL REPORT

for

October - 1962

(Contract NAS 3-2551)

Submitted by

New Product Research of TAPCO

(Attachment to Monthly Progress Report No. 4)

FINANCIAL REPORT

for

Contract NAS 3-2551

for

Period Ending October 31, 1962

	Current Month	Total To Date
TRW Engineering Hours	226	735.5
TRW Costs and Commitments	$ 3,400	$11,443
Subcontractor Costs and Commitments	21,750	43,160
Total	$25,150	$54,603